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1	174	438/633,637,672.ccls. and cmp and particles	USPAT; US-PGPUB	2002/11/07 12:57
2	112	(438/633,637,672.ccls. and cmp and particles) and slurry	USPAT; US-PGPUB	2002/11/07 13:14
3	97	((438/633,637,672.ccls. and cmp and particles) and slurry) and @ad<=20000821	USPAT; US-PGPUB	2002/11/07 13:14
5	63	((438/633,637,672.ccls. and cmp and particles) and slurry) and @ad<=20000821) and (diameter or size)	USPAT; US-PGPUB	2002/11/07 13:14
6	619	438/692,693,687.ccls. and cmp and particles	USPAT; US-PGPUB	2002/11/07 11:08
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11	111	451/36.ccls. and cmp and particles	USPAT; US-PGPUB	2002/11/07 13:06
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25	182	(216/38,89.ccls. and cmp and particles) and slurry	USPAT; US-PGPUB	2002/11/07 13:14
26	161	((216/38,89.ccls. and cmp and particles) and slurry) and @ad<=20000821	USPAT; US-PGPUB	2002/11/07 13:14
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	U	<sup>1</sup> [1]	Document ID	Issue Date	Pages	Title	Current OR
1	<input type="checkbox"/>	<input checked="" type="checkbox"/>	US 20010041446 A1	20011115	18	PRECISION POLISHING APPARATUS FOR POLISHING SEMICONDUCTOR SUBSTRATE	438/692
2	<input type="checkbox"/>	<input checked="" type="checkbox"/>	US 6409781 B1	20020625	13	Polishing slurries for copper and associated materials	51/308
3	<input type="checkbox"/>	<input checked="" type="checkbox"/>	US 6407000 B1	20020618	15	Method and apparatuses for making and using bi-modal abrasive slurries for mechanical and chemical-mechanical planarization of microelectronic-device substrate assemblies	438/693
4	<input type="checkbox"/>	<input checked="" type="checkbox"/>	US 6069083 A	20000530	19	Polishing method, semiconductor device fabrication method, and semiconductor fabrication apparatus	438/693
5	<input type="checkbox"/>	<input checked="" type="checkbox"/>	US 5968239 A	19991019	19	Polishing slurry	106/3
6	<input type="checkbox"/>	<input checked="" type="checkbox"/>	US 5804513 A	19980908	11	Abrasive composition and use of the same	438/693

	Current XRef	Retrieval Classif	Inventor	S	C	P	2	3	4	5	Image Doc. Displayed	PT
1	438/693; 438/745; 438/753; 438/754		TAKAHASHI, KAZUO	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	US 20010041446	<input type="checkbox"/>
2	106/3; 252/79.1; 438/692; 438/693; 51/307; 51/309		Wojtczak, William A. et al.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	US 6409781	<input type="checkbox"/>
3	438/690; 438/692		Hudson, Guy F.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	US 6407000	<input type="checkbox"/>
4	216/38; 216/89; 438/970		Miyashita, Naoto et al.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	US 6069083	<input type="checkbox"/>
5	216/89; 252/79.1; 252/79.2; 252/79.4; 252/79.5; 438/692; 438/693; 51/307		Miyashita, Naoto et al.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	US 5968239	<input type="checkbox"/>
6	106/3; 216/89; 51/308; 51/309		Sakatani, Yoshiaki et al.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	US 5804513	<input type="checkbox"/>

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1	<input type="checkbox"/>	<input checked="" type="checkbox"/>	US 6464740 B1	20021015	19	Reactive aqueous metal oxide sols as polishing slurries for low dielectric constant materials	51/307
2	<input type="checkbox"/>	<input checked="" type="checkbox"/>	US 6354913 B1	20020312	19	Abrasive and method for polishing semiconductor substrate	451/41

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1	106/3; 51/308; 51/309		Towery, Daniel I. et al.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	US 6464740	<input type="checkbox"/>
2	106/3		Miyashita, Naoto et al.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	US 6354913	<input type="checkbox"/>



	U	<sup>1</sup> [1 ]	Document ID	Issue Date	Pages	Title	Current OR
1	<input type="checkbox"/>	<input checked="" type="checkbox"/>	US 6428392 B1	20020806	8	Abrasive	451/36

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1	451/41; 51/307; 51/308; 51/309		Sunahara, Kazuo et al.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	US 6428392	<input type="checkbox"/>

	U	<sup>1</sup> [1 ]	Document ID	Issue Date	Pages	Title	Current OR
1	<input type="checkbox"/>	<input checked="" type="checkbox"/>	US 6403468 B1	20020611	10	Method for forming embedded metal wiring	438/633
2	<input type="checkbox"/>	<input checked="" type="checkbox"/>	US 6380069 B1	20020430	6	Method of removing micro-scratch on metal layer	438/633
3	<input type="checkbox"/>	<input checked="" type="checkbox"/>	US 6350678 B1	20020226	8	Chemical-mechanical polishing of semiconductors	438/633
4	<input type="checkbox"/>	<input checked="" type="checkbox"/>	US 6143656 A	20001107	7	Slurry for chemical mechanical polishing of copper	438/687
5	<input type="checkbox"/>	<input checked="" type="checkbox"/>	US 5770095 A	19980623	16	Polishing agent and polishing method using the same	216/38
6	<input type="checkbox"/>	<input checked="" type="checkbox"/>	US 5607718 A	19970304	40	Polishing method and polishing apparatus	427/97

	Current XRef	Retrieval Classif	Inventor	S	C	P	2	3	4	5	Image Doc. Displayed	PT
1	438/401; 438/675; 438/692		Sugai, Kazumi	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	US 6403468	<input type="checkbox"/>
2	438/672; 438/692		Chen, Hsueh-Chung et al.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	US 6380069	<input type="checkbox"/>
3	438/622; 438/687; 438/690; 438/692; 438/693		Pramanick, Shekhar et al.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	US 6350678	<input type="checkbox"/>
4	438/633; 438/691; 438/692; 438/693		Yang, Kai et al.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	US 6143656	<input type="checkbox"/>
5	216/103; 216/105; 438/633; 438/692; 438/693		Sasaki, Yasutaka et al.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	US 5770095	<input type="checkbox"/>
6	134/2; 216/71; 216/77; 216/89; 438/672; 438/693		Sasaki, Yasutaka et al.	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	US 5607718	<input type="checkbox"/>

US-PAT-NO: 6380069

DOCUMENT-IDENTIFIER: US 6380069 B1

TITLE: Method of removing micro-scratch on metal layer

----- KWIC -----

A method of removing the micro-scratches on a metal layer is described, wherein the metal layer is formed on a barrier layer conformally onto a dielectric layer having a hole thereon, and wherein the metal layer over-fills the hole. The method comprises three chemical-mechanical polishing steps as described hereinbelow. The first chemical-mechanical polishing step is that oxidizing and polishing away the metal layer outside the hole, with a first slurry, wherein the first slurry has a chemical solution and has a plurality of abrasive particles. The second chemical-mechanical polishing step is that polishing away the barrier layer outside the hole, with a second slurry, whereby a plurality of micro-scratches are formed on the metal layer after the barrier layer is chemical-mechanically polished. The third chemical-mechanical polishing step is that buffing the metal layer, with the first slurry, thereby removing the micro-scratches on the metal layer.

The reagent is usually referred to as "slurry". Slurry is a solution mixed with chemicals and abrasive particles. The abrasive particles are extremely hard and have a diameter of about 0.1-0.5 micrometers.

During polishing by a CMP apparatus, the abrasive particles with large size are filtered by a filter in order to reduce the micro-scratches

on the metal surface. However, particles with small size are easily agglomerated into a large size particle. Moreover, while the polishing process stops, even the large size particles deposit. Those large size particles scrape the metal surface and leave micro-scratches thereon, thereby decreasing the yield of the damascene process. Accordingly, we need a method of removing the micro-scratches on a metal surface.

The invention may be incorporated into a method of removing the micro-scratches on a metal layer, wherein the metal layer is formed on a barrier layer conforming onto a dielectric layer having a hole thereon, and wherein the metal layer over-fills the hole. The metal layer is chemical-mechanically polished, to oxidize and polish away the metal layer outside the hole, with a first slurry, wherein the slurry has a chemical solution and has a plurality of abrasive particles. The barrier layer is chemical-mechanically polished to oxidize and polish away the barrier layer outside the hole with a second slurry, whereby micro-scratches are formed on the metal layer. The metal layer is chemical-mechanically polished again, to buff the metal layer, with the first slurry, thereby removing the micro-scratches on the metal layer.

The metal layer is preferably buffed for about 1 to about 30 seconds with the slurry. Moreover, the buffing slurry has a preferable chemical concentration less than that of the slurry used for oxidizing and polishing away the metal layer outside the hole. The chemical concentration is lowered for slowing down the buffing removal rate. However, alternatively, the buffing removal rate can also be slowed down by reducing the number of the abrasive

particles from the

slurry. Another method of slowing down the polishing removal rate is to adjust the parameters of the CMP apparatus.

In the first polishing step 202, a first slurry oxidizes the metal layer 106 outside the hole 102 while the applied mechanical force along with friction polishes away the oxidized metal. After the oxidized metal is polished away from the filed regions, a second slurry is introduced that removes the barrier layer 104 (e.g. Ti and TiN) outside the hole 102. This second polishing step 204 continues until the dielectric layer 108 is slightly eroded, to ensure that the field regions are cleaned of all residual metal.

Still referring to FIG. 2 and FIG. 1, the metal CMP process further involves a buffing step (a third polishing step) 208 oxidizing and polishing away a thin thickness of the first-polished metal layer 106 on the surface by using the first slurry. The buffing step 208 removes the micro-scratches that can increase the yield of the dual damascene processing. Verified by experiments, applying this buffing step 208 on an 8 inches wafer reduces the number of the defects from about several hundreds to about less than one hundred. At the same time, the micro-scratches on the metal layer 106 are also removed by this buffing step 208.

Because the micro-scratches are very shallow and have an average depth of only about several nanometers, the first slurry to the metal buffing step 208 preferably has a chemical concentration lower than that to the first polishing step 202, for slowing down the metal-buffing rate. Alternatively, the abrasive particles from the first slurry to the metal buffing step 208 have a number

less than that to the first polishing step 202. Such first slurry to the metal buffing step 208 has a preferable abrasive-particle concentration of about 0-50%, and most preferably of about 10%. The concentration decrease or the abrasive-particle reduction facilitates the control of the metal buffing step 208.

Before the metal layer 106 is buffed and after the first and the second polishing steps 202, 204, another polishing step may be needed for buffing of the dielectric layer. As shown in FIG. 3, which is another flow chart illustrating a metal CMP process according to another preferred embodiment of this invention. In this flow chart, the additional polishing step 206 buffs the dielectric surface 108 with a third slurry. The steps 202a, 204a, 208a are substantially the same as the steps 202, 204, 208 of FIG. 2.

chemical-mechanically polishing the metal layer, to oxidize and polish away the metal layer outside the hole, with a first slurry, wherein the slurry has a chemical solution and has a plurality of abrasive particles;

chemical-mechanically polishing the barrier layer to oxidize and polish away the barrier layer outside the hole with a second slurry; and

chemical-mechanically polishing the metal layer, to buff the metal layer, with the first slurry.

3. The method according to claim 1, wherein the slurry used for buffing the metal layer has a chemical concentration less than that of the slurry used for oxidizing and polishing away the metal layer outside the hole.



4. The method according to claim 1, wherein the abrasive particles from the slurry to buff the metal layer have a number less than that to polish away the metal layer outside the hole.

5. The method according to claim 1, wherein the method according to claim, wherein the slurry to buff the metal layer has a abrasive-particle concentration less than about 50%.

6. The method according to claim 5, wherein the method according to claim, wherein the slurry to buff the metal layer have a abrasive-particle concentration of about 10%.

chemical-mechanically polishing the metal layer, to oxidize and polish away the metal layer outside the hole, with a first slurry, wherein the first slurry has a chemical solution and has a plurality of abrasive particles;

chemical-mechanically polishing the barrier layer, to polish away the barrier layer outside the hole, with a second slurry; and

chemical-mechanically polishing the metal layer, to buff the metal layer, with the first slurry.

12. The process according to claim 10, wherein the first slurry used for buffing the metal layer has a chemical concentration less than that of the first slurry used for polishing away the metal layer outside the hole.

13. The process according to claim 10, wherein the abrasive particles from the first slurry to buff the metal layer have a number less than that to polish away the metal layer outside the hole.

14. The process according to claim 10, wherein the first slurry to buff the metal layer has a abrasive-particle concentration less than about 50%.

15. The process according to claim 14, wherein the first slurry to buff the metal layer have a abrasive-particle concentration of about 10%.

18. The process according to claim 10, further comprises chemical-mechanically polishing the dielectric layer, to buff the dielectric layer, with a third slurry, before the metal layer is buffed and after the barrier layer is oxidized and polished away.

US-PAT-NO: 6409781

DOCUMENT-IDENTIFIER: US 6409781 B1

TITLE: Polishing slurries for copper and associated materials

----- KWIC -----

A chemical mechanical polishing slurry and method for using the slurry for polishing copper, barrier material and dielectric material that comprises a first and second slurry. The first slurry has a high removal rate on copper and a low removal rate on barrier material. The second slurry has a high removal rate on barrier material and a low removal rate on copper and dielectric material. The first and second slurries at least comprise silica particles, an oxidizing agent, a corrosion inhibitor, and a cleaning agent.

The present invention is directed to a chemical mechanical polishing slurry comprising a first slurry, which has a high removal rate on copper and a low removal rate on barrier material and a second slurry, which has a high removal rate on barrier material and a low removal rate on copper and the associated dielectric material. The first and second slurries comprise silica particles, an oxidizing agent, a corrosion inhibitor, and a cleaning agent. Also disclosed as the present invention is a method for chemical mechanical polishing copper, barrier material and dielectric material with the polishing slurry of the present invention. As will become apparent from the discussion that follows, the stable slurry and method of using the

slurry provide for removal of material and polishing of semiconductor wafer surfaces with significantly no dishing or oxide erosion, with significantly no surface defects and good planarization efficiency, and produce a copper surface with minimal corrosion tendency post-polish.

FIG. 6 is a transmission electron micrograph (TEM) showing 13 nm silica particles of the present invention.

FIG. 7 is the size distribution of 13 nm silica particles of the present invention determined with Coulter N4 Plus particle analyzer.

Turning now to the composition of the CMP slurry, generally the first and second slurries comprise silica particles, an oxidizing agent, a corrosion inhibitor, and a cleaning agent. The chemistry of the first and second slurries should be stable and have a pH in the range of about 2 to 5. The first and second slurries may contain potassium or ammonium hydroxide in such amounts to adjust the pH to a range of about 2 to 5.

The silica particles of the first and second slurries can be precipitated. The precipitated particles usually range from about 3 to 100 nm in size and can be spherical. An alternative to precipitated silica particles in the first slurry is fumed silica. Generally, the fumed silica has a mean particle size of less than 700 nm.

Alternatively, and more preferred is to use colloidal silica particles of the type described. The colloidal silica particles can range from about 3 to 100 nm in size, and can be spherical. Preferentially, when the first and second slurries employ spherical colloidal particles, the

particles should have a narrow size distribution. More specifically, about 99.9% of the spherical colloidal particles should be within about 3 sigma of a mean particle size with negligible particles larger than about 500 nm.

The first slurry, thus, can employ either precipitated spherical silica particles in the size range of 3 to 100 nm, or fumed silica with mean particle size less than about 700 nm. These particles coupled with the iodate chemistry allows the first slurry to achieve high copper removal rate but minimal barrier material removal rate. Colloidal silica, with a narrow size distribution, minimizes micro scratch defects and provides superior removal rates on barrier materials, greater than about 1000 .ANG./min, and low removal rates for copper for the second slurry. Further, spherical silica abrasives with a mean size of less than about 100 nm provide very good planarization efficiency.

The pH, oxidizing agents, modifying agents, abrasive particle composition and size distribution, and weight percent were evaluated to establish a baseline for removal rates and selectivity.

Precipitated silica mean particle sizes of 8 nm, 20 nm, and 70 nm were tested.

The fumed silica particle size tested was less than 700 nm.

The optimum CMP slurry, including the first and second slurry, had a precipitated silica mean size of less than about 100 nm. The optimum fumed silica abrasive mean size for the first slurry is less than about 700 nm. The optimum CMP slurry formulations contain 1-10% precipitated silica, or fumed silica for the first slurry.

Further, different types of abrasive particles were studied to maximize the removal and selectivity characteristic of the slurry. Precipitated silica abrasives, with mean size distributions of 4 nm, 8 nm, 13 nm, 20 nm and 70 nm were tested. FIG. 6 shows a TEM picture of 13 nm slurry. The size distribution of these particles is presented in FIG. 7. Fumed silica, with a mean particle size of less than about 700 nm, was also evaluated. All of these mean size distributions can be used to achieve effective polishing rates and selectivities for the first and second slurries.

Several formulations of the first slurries were prepared. The characteristics of these formulations are described in Table 2. The first slurry is optimally comprised of formula 5, for colloidal silica particles, and formula 19 for fumed silica particles. Thus, the first slurry is preferentially comprised of 1-10% colloidal silica with particle size 3 to 100 nm, or 1-5% fumed silica with mean particle size of less than about 700 nm. Further, the active chemistry for the optimum first slurry is about 1-12% potassium iodate (KIO<sub>3</sub>, formed by reaction of HIO<sub>3</sub> with KOH), which is used as the oxidizing agent for the copper, about 0-5% concentrated inorganic acid as a copper activating agent, and 0-2% iminodiacetic acid (IDA) as the copper corrosion inhibitor and cleaning agent.

As can be seen from Table 2, all of the first slurry formulations of the present invention were effective in achieving acceptable copper removal rates, and semiconductor wafer surfaces of high quality. Thus, the first slurry is preferentially comprised of 1-10% colloidal silica with particle size of less than about 100 nm.

silica particles selected from the group consisting of:

19. The chemical mechanical polishing slurry of claim 1, wherein said first and second slurries contain precipitated silica particles.

20. The chemical mechanical polishing slurry of claim 19, wherein said precipitated silica particles are about 3 to 100 nm in size.

21. The chemical mechanical polishing slurry of claim 19 wherein said precipitated silica particles are spherical.

22. The chemical mechanical polishing slurry of claim 20 wherein said precipitated silica particles are spherical.

23. The chemical mechanical polishing slurry of claim 2 wherein said silica particles for said first slurry are fumed silica.

24. The chemical mechanical polishing slurry of claim 23, wherein said fumed silica particles have a mean particle size of less than 700 nm.

25. The chemical mechanical polishing slurry of claim 2 wherein said silica particles for the first slurry are colloidal silica particles.

26. The chemical mechanical polishing slurry of claim 25 wherein said colloidal silica particles are about 3 to 100 nm in size.

27. The chemical mechanical polishing slurry of claim 25 wherein said colloidal silica particles are spherical.

28. The chemical mechanical polishing slurry of claim 26 wherein said colloidal silica particles are spherical.

29. The chemical mechanical polishing slurry of claim 27

wherein id particles  
have a size distribution in a range of from about 3 nm to 100 nm.

30. The chemical mechanical polishing slurry of claim 27 wherein about 99.9% of said particles are within about 3 sigma of a mean particle size with particles larger than 500 nm.  $\leq 0.1\%$ .

33. The chemical mechanical polishing slurry of claim 31 wherein said colloidal silica has a particle size of about 3 to 100 nm.

34. The chemical mechanical polishing slurry of claim 32 wherein said fumed silica has a mean particle size of less than 700 nm.

38. The chemical mechanical polishing slurry of claim 37 wherein said colloidal silica has a particle size of less than 100 nm.

weight % 1-10 colloidal silica particles 0.1-1.0  
potassium iodate 0-2  
iminodiacetic acid